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(19) (CA) **APPLICATION FOR CANADIAN PATENT** (12)

(54) Method of Treating Materials at Low Temperatures

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(73) Same as inventor

(57) 9 Claims

Notice: The specification contained herein as filed

Canada

METHOD OF TREATING MATERIALS AT LOW TEMPERATURES

The present invention relates to treating materials at low temperatures and more specifically to a method of enhancing the performance of materials such as tool steel, plastics and natural fibres by subjecting them to relatively cold temperatures for a specific time/temperature program.

It is known that dry cryogenic treatment of metals and selected organic materials improves the properties of these materials. Reference is made to my Canadian Patent No. 1,143,581, issued March 29, 1983, which discloses a process and apparatus for cryogenic treatment of materials. The process discloses a single cooling step to a low temperature, generally the temperature of nitrogen or in some cases helium retaining the material at that temperature for some time and then allowing a slow warming of the material to avoid stresses occuring in the material.

I have now found that I can provide a more economical method of treating materials at low temperatures wherein I utilize carbon dioxide for an initial cooling step, cooling the materials down to about -70°C, followed by a second cooling step with a short intermediate stage wherein the temperature of the materials is lowered to about -190°C, which is the approximate temperature of liquid nitrogen at atmospheric pressure.

The present invention provides a method of treating materials at low temperatures, comprising the steps of first cooling the materials in an enclosed chamber by lowering the temperature in the chamber slowly from ambient down to about -70°C, maintaining the materials in the chamber at about -70°C for at least three days, second cooling the materials further by lowering the temperature in the chamber slowly

down to about -190°C, maintaining the materials in the chamber at about -190°C for about one day, first raising the temperature in the chamber slowly to about -70°C, maintaining the materials in the chamber at about -70°C for about two days, and second raising the temperature of the chamber slowly so that the materials warm up to ambient temperature.

In a drawing which illustrates an embodiment of the invention, the Figure is a graph showing the temperature against time for a typical low temperature program.

In Canadian Patent No. 1,143,581 is disclosed a cryogenic treatment apparatus which, while suitable for carrying out the process disclosed therein, is also suitable for carrying out the method of the present invention. An enclosed chamber is provided which may be similar to that shown in Canadian Patent 1,143,581. The chamber has a door or lid that allows materials to be placed therein, and the chamber closed. The chamber is insulated and has provision for introducing liquid gas to cool the chamber as the liquid evaporates. Provision is made to control the cooling rate inside the chamber, maintain the temperature at a predetermined value within the chamber and then to slowly warm the chamber to bring it back to ambient temperature at a specified rate.

By introducing a two stage cooling and retaining the material at a low temperature for a longer time then disclosed in my previous Canadian patent, I have found that improved properties occur in tool steels and other materials. Initially, the materials are placed in an enclosed chamber and by utilizing liquid carbon dioxide the temperature of the materials in the chamber is slowly lowered to about -70°C. The pressure in the chamber is generally kept at atmospheric although higher or lower temperatures may be used. As in my previous Canadian patent, in one embodiment the cooling rate of materials in the chamber is about 0.5°C per minute from ambient down to

about -70°C. It is important that a strict control occur in the cooling to avoid warping or other stress related damage occurring in the material.

In the embodiment disclosed herein the temperature drop for the first cooling stage from ambient to -70°C takes approximately 20 hours, equivalent to about 5° per hour which is less than 0.1° per minute. The temperature of the materials in the chamber then remains at about -70°C for at about three days, and as shown in the Figure for 90 hours. During this time the austenite in steel transforms to martensite which assists in promoting aging and homogenization of the metal.

The temperature in the chamber is then dropped in a second cooling stage to about -190°C by the addition of liquid nitrogen or, if colder temperatures are desired, liquid helium may be used which has a temperature of around -268°C at normal atmospheric pressure conditions.

The cooling rate is preferably less than 0.5°C per minute and in the embodiment shown in the Figure, the temperature drops from -70° to -190°C in approximately 20 hours which is at approximately the same rate as the first cooling stage, namely about 0.1° per minute.

The temperature of the materials is maintained at about -190°C when liquid nitrogen is used which precipitates carbides and promotes homogenization of the metal components and granular structure. Furthermore, retention at this temperature assists in stress relief and accelerates aging of the metal.

Following about one day, or in the specific example shown in the Figure, approximately 20 hours, the temperature of the materials inside the enclosure is slowly raised in the first warming stage under controlled conditions to about -70°C. In the example shown in the Figure the time for the

temperature to raise from -190° to -180°C is 30 hours which is slightly faster than 3.5° per hour. The temperature of the material in the chamber is retained at about -70°C for at least 30 hours and in the example shown in the Figure, 40 hours, and is then allowed to slowly warm up from -70°C to ambient temperature in the second warming stage. The rate of warming up in the second warming stage is preferably not greater than 0.25°C per minute and in the example shown in the Figure takes 20 hours which is approximately 5° per hour. The rise in temperature is controlled strictly to ensure that no stress damage occurs to the material. One method of controlling cooling and warming is disclosed in my Canadian Patent 1,143,581.

The total time for the process as shown in the Figure is 240 hours, or ten days. The second warming step from -70°C to ambient temperature occurs in preferably about one day, although 20 hours is shown in the Figure.

The method of the present invention may be used with tools used in sawmills, specifically debarking equipment made of tungsten carbide, chip-n-saw knives, chipper knives, planer knives. Files and grindstones may also be treated, as well as razor blades. Other materials than steel, such as plastics and natural fibres, also have improved performance after this treatment, where durability properties are improved as well impact properties.

With regard to tests conducted in sawmills, it was found that debarking knives lasted three and one half times longer when treated to the cryogenic treatment as disclosed in Canadian Patent 1,143,581 than untreated knives. The same knives had a life of over four times the untreated life when treated by the method of the present invention. Chip-n-saw knives had a life increase of three for the cryogenic treatment and this increased to a life of three and one half times after the treatment of the present invention. Chipper knives had an increased life four times greater than

untreated for both the cryogenic treatment and for the treatment of the present invention. Planer knives have an increased life of two and one half times greater than untreated knives for the cryogenic treatment and this increased to four and one half times greater for the treatment of the present invention. Files, grindstones and razors had substantially the same life which was greatly increased over the untreated products, when either cryogenically treated or treated by the method of the present invention.

Various changes may be made to the embodiment shown herein without departing from the scope of the present invention which is limited only by the following claims.

The embodiments of the present invention in which an exclusive property or privilege is claimed are defined as follows:

1. A method of treating materials at low temperatures, comprising the steps of:

first cooling the materials in an enclosed chamber by lowering the temperature in the chamber slowly from ambient down to about -70°C,

maintaining the materials in the chamber at about -70°C for at least three days,

second cooling the materials further by lowering the temperature in the chamber slowly down to about -190°C,

maintaining the materials in the chamber at about -190°C for about one day,

first raising the temperature in the chamber slowly to about -70°C,

maintaining the materials in the chamber at about -70°C for about two days, and

second raising the temperature of the chamber slowly so that the materials warm up to ambient temperature.

2. The method of treating materials according to Claim 1 wherein the first cooling occurs by the introduction of liquid carbon dioxide into the chamber.

3. The method of treating materials according to Claim 1 or Claim 2 wherein the second cooling occurs by the introduction of liquid nitrogen into the chamber.

4. The method of treating materials according to any of Claims 1, 2 or 3 wherein the first cooling from ambient down to about -70°C occurs in about 20 hours.

5. The method of treating materials according to any of Claims 1, 2 or 3 wherein the second cooling from about -70°C down to about -190°C occurs in about 20 hours.

6. The method of treating materials according to any of Claims 1, 2 or 3 wherein the first raising the temperature in the chamber from about -190°C to about -70°C occurs in about one day.

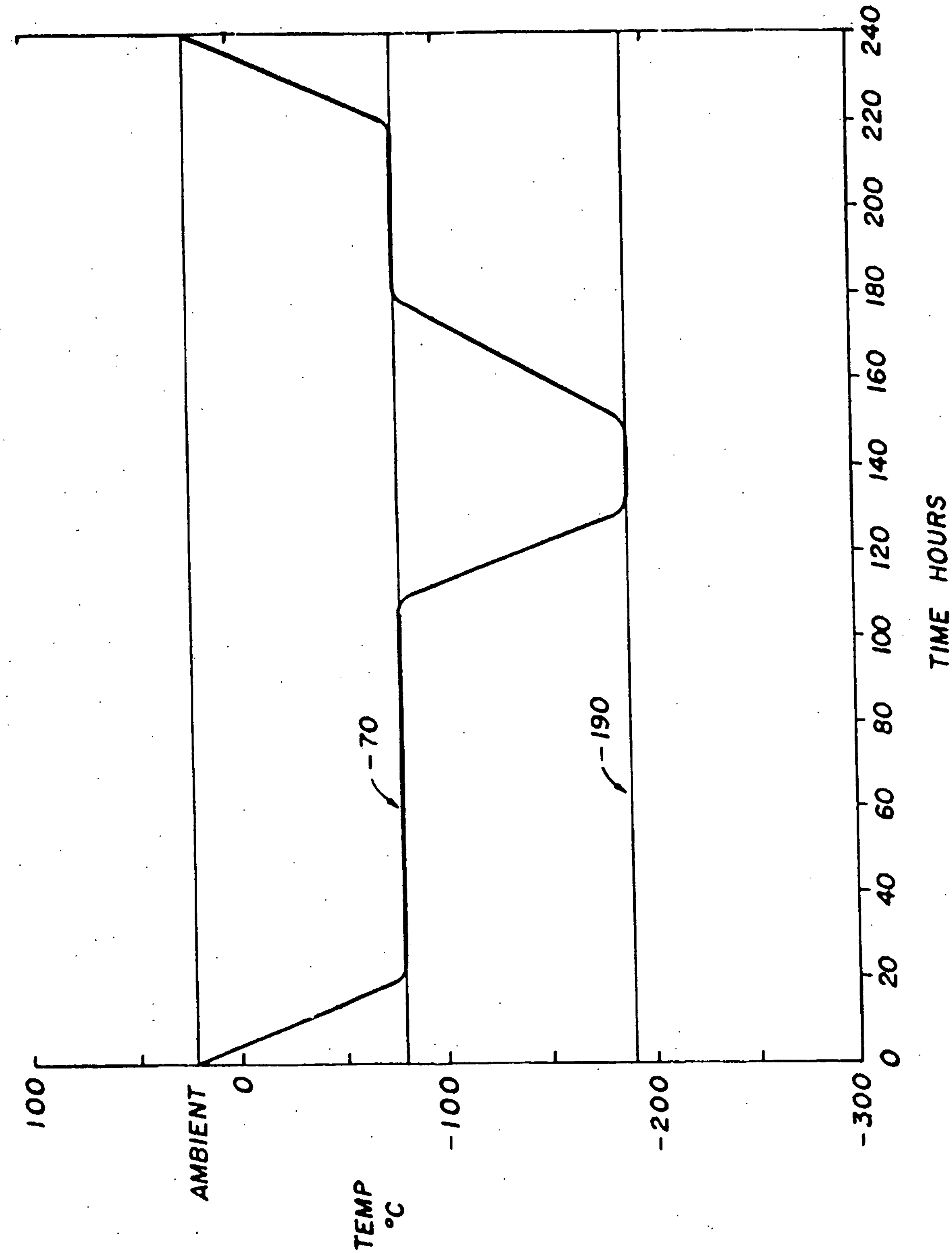
7. The method of treating materials according to any of Claims 1, 2 or 3 wherein the second raising the temperature from about -70°C to ambient occurs in about one day.

8. The method of treating materials according to any of Claims 1, 2 or 3 wherein the method occurs in about ten days.

9. The method of treating materials according to Claim 1 or Claim 2 wherein the second cooling occurs by the introduction of liquid helium into the chamber.

ABSTRACT OF THE DISCLOSURE

Materials such as tool steel, plastics and natural fibres are treated at low temperatures to enhance their performance. An economical process provides for first cooling the material down to about -70°C for a predetermined time followed by an intense cooling step down to about -190°C for a short duration. The materials are allowed to warm up in two stages pausing between stages at about -70°C. The specific time/temperature program utilizes liquid carbon dioxide and liquid nitrogen and may be varied for the materials to be treated.



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